INVESTIGATIONS OF MEDICINAL PLANTS MEDIATED NANOMATERIALS AS ELECTRODE AND POLYMER ELECTROLYTE FOR SUPERCAPACITOR APPLICATIONS

A SYNOPSIS

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1. INTRODUCTION

The international union for conservation of nature and the world wildlife fund announced that species of flowering plants between 50,000 to 80,000 are utilized for medicinal purposes across the entire globe. These were initially utilized in crude forms like powders, tinctures, teas, poultices, etc. Subsequently, herbal medicine from the roots, leaves, flowers or bark, and seeds of the plant is referred to the treatment of many conditions of diseases such as eczema, asthma, premenstrual syndrome, diabetes, rheumatoid arthritis, jaundice, cardiovascular diseases, iron overloaded linked to disease, menopausal symptoms, migraine, irritable bowel syndrome, chronic fatigue and cancer [1]. Following this, the isolation of active compounds from medicinal plants started beginning in the 19^{th} century leading to the synthetic forms of tablets in the herbal medicine markets. In the continuation process, *solanaceae* family plants are preferable for bio-compounds under the COVID-19 and also recent diseases Monkeypox, Influenza A (H1N1), Ebola virus and cancer. Recently, plant extracts have found applications as effective and eco-friendly, reducing agents for the synthesis of nanoparticles (NPs). Therefore, green synthesized NPs are useful for innovations in their various applications. To address the issues of low-maintenance and cost-effectiveness researchers are devoted to improving the clean and renewable energy from wind, tide, sun, and biomass [2]. Hence renewable energy sources are gaining importance day by day. Most of the existing energy storage technologies are thermal, chemical, thermochemical, magnetic, hydrogen, flywheel, pumped, and compressed air energy storage [3]. Among these techniques, electrochemical energy storage has been considered one of the highly successful ways to convert chemical energy into electrical energy. This storage set-up has large theoretical

efficiency and solid-state electrodes, which are equip the energy, and power density. In electric power future, Supercapacitors (also known as ultracapacitors) are a big part of the electrochemical system to need to store and release large amounts of electrical energy very quickly. Although there are many highlights in green synthesis NPs, it is aimed at how medicinal plants can be used in the booming field of Supercapacitor because heteroatoms in organic material boost up the electrochemical performance. Therefore, this research work deals with the synthesis of nanomaterials as electrodes and polymer electrolyte for Supercapacitor, using leaf extract from the medicinal plants *Datura metel L*. and *Withania somnifera*.

2. LITERATURE REVIEW

The existing literature review is done on the following topics.

- Solanaceae family of plants
- Collection of data regarding *Datura metel L.* and *Withania somnifera*
- Nanoparticles synthesis and electrochemical performance of nanomaterials prepared from the *Datura metel L.* and *Withania somnifera*
- Plant-mediated synthesis of Cu_2O NPs, Ag NPs, Ag/NiO nanocomposite and test their electrochemical performance
- Sodium alginate used as a polymer electrolyte

Recently, Tripathi et al. [4] suggested the *Withania sonmnifera* plant with potential application in the treatment of cancer and SARS-COV-2.

Yulizar et al. [5] employed the alkaloids available in the leaf extract of *Datura metel L.* as a capping agent for the synthesis of CeO_2 NPs. Prasad et al. [6] used root extract of *Withania somnifera* for the synthesis of ZnO NPs. They prepared ZnO NPs with help of carbonyl, amine, and hydroxyl groups from the aqueous extract of zinc acetate. The scanning electron microscopy analysis shows the reducing ability of *Withania somnifera* root extract.

Raj et al. [7] tested the Supercapacitor performance seed pot of *Datura metel* as active electrodes for the derivation of N, S co-doped activated mesoporous carbon. They obtained specific capacitance of 340 F/g at a current density of 1 A/g and maintained the cyclic retention of 95.24 % after the 3000 cycles, these noticeable results are achieved by the mesoporous architecture due to the N, S functionality. Raveesha et al. [8] reported electrochemical properties of *Withania somnifera* based on the synthesis of MgO NPs. Plant extract based synthesis ensures a conductive metal-organic framework that increases the energy device performance. For instance, Siva Shalini et al. [9] has attained a maximum specific capacity as high as 606.2 C/g and cyclic stability of about 131.14 % after 5000 cycles at a current density of 5 A/g. In their asymmetric Supercapacitor that employed a conductive metal-organic framework of Ag. Sundari et al. [10] analyze the (asymmetric) Supercapacitor performance of NiO NPs prepared through *ficus-indica* leaf extract and reported surface faradaic provides the specific capacitance of ~ 644 F/g at a scan rate of 10 mV/s. Zhou et al. [11] synthesized Ag/NiO structure by the hetero-MOF (Metal organic framework) for Supercapacitor applications. The author reported a specific capacitance of 1480 F/g at the current density of 0.6 A/g in 1 mole of KOH electrolyte with 85 % of capacity retention observed even after 3000 cycles at the current density of 5 A/g.

Besides plant extract based synthesis of NPs, we also focus on the plant extract incorporated polymer (sodium alginate) based separators. Song et al. [12] have evaluated the thermal stability, electrolyte wettability and tensile strength of sodium alginate/attapulgite separator. Electrochemical lithium cells assembled using this separator improved the battery safety from the thermal point of view.

From the above literature survey, we understand that NPs synthesis from leaf extract of *Datura metel L.* and *Withania somnifera* is yet to be explored.

3. RESEARCH GAP

Today, everyone is thinking about sustainable clean energy for everyday life because crude oil, petrol prices are hiked on progressively, which is drives the global economy. Therefore, we will need to move away from fossil fuels. Hence, wide array of technological approaches are exist to resolve the issues of energy crisis such as thermal, mechanical, hydrogen and pumped hydropower. But, we need to environmentally friendly and sustainable alternative energy resources because of energy and environment are two of critical challenges. This could be made possible by renewable sources such as wind, solar, water, geothermal and biomass. The energy generated by the mentioned renewable resources stored in fuel cells, photovoltaic cells, capacitors and batteries. But, still does not efficiently meet the demand of human needs. Hence energy storage has become an important topic all over the globe. Batteries and capacitors are doing similar job i.e. energy storage. Batteries are can hold large amount of energy, but they take hours to charge up and capacitors charges almost instantly but store only tiny amount of energy. To overcome the issues of batteries and capacitors to move towards on Supercapacitor, here can store a charge thousands, millions or even billions of times bigger than batteries, capacitors and it has the ability to store and release energy very quickly. This Supercapacitor consists of two parts, one is electrode and another is electrolyte. By choosing these two properly, it can achieve its high performance. Therefore, various research efforts are underway in acceleration of electrode preparation. In recent years, Singhbabu et al. [13] prepared RGO-NiO electrode using *Aeschynomene Aspera.* Shaheen et al. [14] prepared Co_3O_4:NiO electrode using *Euphorpia cognata*. This kind of research work makes intention to us, Preparation of electrode nanomaterials using medicinal plants because India is one of the rich medicinal plants country in this world, which include 75000 medicinal plants. Till date, there are minimal amount of research work has done in energy storage application utilizing medicinal plants. In particular, the Solanaceae family plants usage of nanomaterials synthesis is very low. However, the nanoparticles synthesized by *Datura metel L.* and *withania*

somnifera using Cu_2O, NiO and Ag NPs as electrodes in Supercapacitor detailed manner has not yet done so far. Therefore, this research work has been made to attempt the synthesis of nanomaterials mediated by medicinal plants through *Datura metel L.* and *Withania somnifera* used as electrodes and also *Withania somnifera* incorporated polymer film as electrolyte in Supercapacitor application and their preparations are shown in Figure 1 and 2.

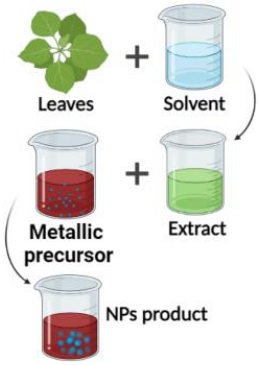

Figure 1 Common route of green synthesized nanoparticles

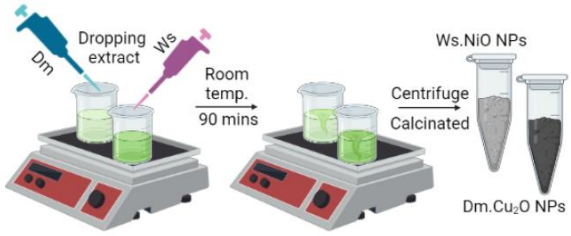

Figure 2 Facile green synthesized metal oxide nanoparticles in diagram

4. RESEARCH PROBLEM

High-efficiency electrochemical device is one of the most important needs of today, and the role of electrodes and electrolytes is immense in its development. Therefore, the properties of these electrodes depend on the properties of the materials from which they are made. Especially the surface-to-volume ratio and porous structure properties make it very suitable for redox mechanism (diffusion of ions/electrons, adsorption/desorption). So both these properties are easily possible in NPs. Although various methods are in practice to produce these NPs, green synthesis remains the most popular nowadays. Because bioactive compounds like flavonoids, polyphenols and alkaloids, etc.,

in the bacteria, fungi, and plant extracts plays pivotal role with excellent reducing agents, provide stability, and are non-toxic. Apart from green synthesis, sodium citrate, sodium borohydrate, and alcohol, which can be used in other synthesis processes like physical and chemical methods namely arc discharge, thermal decomposition, irradiation, co-precipitation, solvothermal, and hydrothermal to reduce the metal precursor, are toxic and can cause adverse human health conditions and above mentioned each method has own limitation on some point view. Non-toxic, harmless nanoparticles are therefore needed for a wide range of applications. One of the energy source technologies, Supercapacitor, is developing as a renewable energy type in the future, so clean energy storage is considered essential. Therefore, nanoparticles produced by green synthesis are gaining importance in Supercapacitor applications. Ag NPs are primarily in this green synthesis because of the ease of reduction and small size, specific favourable morphologies. All these properties lend themselves well to electrochemical energy storage mechanisms. Also, its good electrical conductivity plays a major role in promoting redox behaviours. Moreover, NiO NPs with higher theoretical capacity and quantum confinement properties can provide better performing electrodes. Meanwhile, CuO nanoparticles, which can easily be given a variety of morphologies (spherical, oval, cubic, pillar, Hexagonal, and marigold flower like structure) by their nature, can also provide well performed electrodes. Because NPs with different morphologies are could easily maximize the redox mechanism. Thus, marigold flower like - shaped Cu_2O NPs, spherical Ag NPs and mixed spherical, hexagonal Ag/NiO nanocomposites have shown significant specific capacitance. However, when two nanoparticles with different properties are brought together, the two combine to form summarized results. Therefore, the electrochemical properties of the Ag/NiO nanocomposite are likely to be enhanced. As mentioned above, when nanomaterials of Cu_2O NPs, Ag NPs and Ag/NiO nanocomposite are formed through green synthesis their electrochemical properties are increased due to the phytochemicals (O, C, S, N) attached to it help a lot. Therefore, in order to know the electrochemical properties caused by these phytochemicals

electrolyte was developed using plant extract of *Withania somnifera* because polymer electrolyte is an important part of the electrochemical devices.

5. OBJECTIVE OF THE RESEARCH WORK

In the next few years, our fossil fuel powered vehicles and home heating will have to switch to electric power. But it also suffers from a major drawback. The idea is to store this electrical energy and make it available for long-term use which is effectively done by Supercapacitor. However, to increase its efficiency and meet the demand for clean energy, its main component electrodes are produced with the help of biomass, which gives more electrochemically active sites. Therefore, there is a need for nanoparticles that can be produced by simple, inexpensive, and environmentally friendly green synthesis. All these reasons motivate us to develop medicinal plants (*Datura metel L. and Withania somnifera*) mediated nanoparticles as an electrode and *Withania somnifera* incorporated polymer film as electrolyte in Supercapacitor application. Therefore the following processes have been carried out in this research.

- Literature review of medicinal plants, collection of data on their efficacy with PubMED, PubChem.
- Screening of Solanaceae family plants for nanomaterials synthesis.
- Green synthesis of Cu_2O NPs and Ag NPs using *Datura metel L*.
- Preparation of Ag/NiO nanocomposite at different concentrations using *Withania somnifera* by green synthesis method.
- To understand the physicochemical properties such as optical, functional group, structural, morphological and elemental composition of Cu_2O NPs, Ag NPs and Ag/NiO nanocomposite to be analyzed through Ultraviolet-Visible (UV-Vis), Fourier Transform Infrared (FT-IR), X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) investigations.

- To study morphology, chemical composition, thermal and magnetic properties of Ag/NiO by High Resolution Transmission Electron Microscopy (HR-TEM), X-ray Photoelectron Spectroscopy (XPS), Differential Thermal Analysis (DTA)/Thermogravimetric Analysis (TGA) and (Vibrating Sample Magnetometer (VSM) analysis, respectively.
- To evaluate the Supercapacitor performance of as-synthesized Cu_2O NPs, Ag NPs, and Ag/NiO nanocomposite are made as electrodes.
- To realize the importance of different morphologies of nanoparticles and phytochemical performance in electrochemical analysis.
- Fabrication of symmetric and asymmetric Supercapacitor devices by well-performing nanoparticles and determination of power and energy density.
- Meanwhile, *Withania somnifera* is one of the Solanaceae family plants selected for this research was used to prepare the polymer electrolyte and test its performance.

6. SCOPE OF THE RESEARCH WORK

Engineered nanoparticles and their technological implications are already generating breakthrough ideas in industrial processes by improving optical, electrical, magnetic, and biological properties. Green synthesis of NPs using plant extracts of *Datura metel L.* and *Withania somnifera* has yielded several enhanced benefits. Polyphenol, flavonoids, and alkaloids are the main source of above mentioned plant extract, which are used for the preparation of metal and metal oxide NPs through the reduction mechanism. Moreover, these are could be acting as stabilizing, capping agents for the reduction of metal precursors. Therefore, heteroatoms such as O, C, S, P, and N etc., are found in the prepared NPs from medicinal plant material leading to the performance of

Supercapacitor. The extension of internal characteristics studies done by the simulation of the algorithm will help for the complete analysis of plant-mediated NPs on Supercapacitor material in the upcoming days. The phytoconstitutions of *Datura metel L.* and *Withania somnifera* boost the specific capacitance of the electrodes and responded to the electrochemical performance of polymer electrolytes. This research work could be identified as an active electrode and electrolyte material for Supercapacitor devices.

7. Organization of the thesis

The organization of the thesis work is carried out by a flow chart shown in Figure 3.

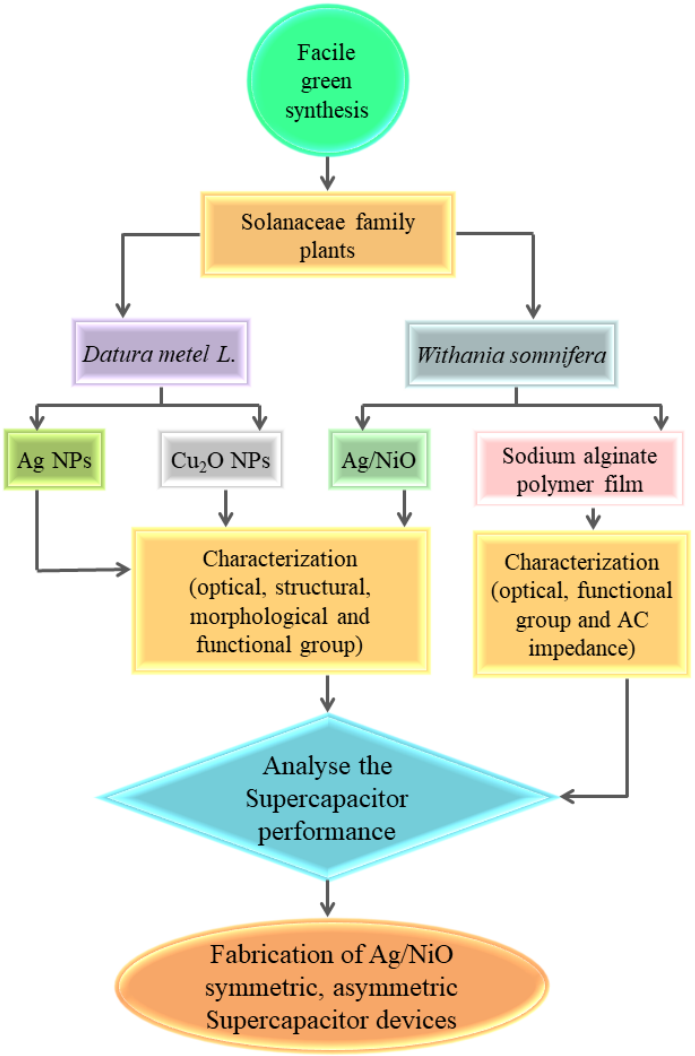

Figure 3 Sketch of the thesis work

Chapter 1 – Introduction

Chapter 1 discusses the background of the medicinal plant as an alternative source for energy storage applications due to the demand for fossil fuels. Concerning this, the importance of medicinal plants, the necessity of phytochemicals, and the focus of this research work on Solanaceae family plants are discussed. Due to the environmentally friendly nature and cost-effectiveness of NPs management possible in the green synthesis method, hence metal oxide (NiO, CuO) NPs and metal (Ag) NPs general discussion about their properties are discussed. The detailed description of energy storage devices such as a capacitor, Supercapacitor, and their classification namely Electric Double Layer Capacitor (EDLC), Pseduocapacitor, hybrid Supercapacitor, and assembling of Supercapacitor is deliberate. General discussion about polymer electrolytes in an electrochemical capacitor, categorization of polymer electrolytes namely solid, gel, composite electrolyte than sodium alginate as a biopolymer electrolyte, its general properties and merits, demerits are summarized.

Chapter 2 – Literature review

Chapter 2 discuss the literature review of research work entitled "medicinal plant-mediated nanoparticles as an electrode and conducting polymer electrolytes for Supercapacitor applications" In this chapter, discussed the outline of the literature review and reveals the account of gained importance of several *Solanaceae* family plants in terms of pharmaceutical properties in different applications on recent days. As in the member of this family, *Datura metel L.* and *Withania somnifera* species exhibit the various biological activities analyzed in recent reports are discussed. Specifically, isolated several bio compounds from these plants and its employment in COVID-19 diseases are also noted. Afterward, the frame of nanoparticles synthesized with help of both plants and their role in Supercapacitor applications done by a recent researcher discussed. Preparation of Cu_2O NPs,

Ag NPs and Ag/NiO nanocomposite using leaf extracts and its Supercapacitor performance are examined. The recent reports of sodium alginate as an electrolyte are discussed. This chapter also covered the identification of the research gap and its existing problem, objectives, and scope of this research work are discussed.

Chapter 3 – Materials and Methods

In this chapter 3, materials are used to carry out the research and green synthesis methodology are Cu_2O NPs, Ag NPs using *Datura metel L*. leaf extract [DMLE] and Ag/NiO nanocomposite via sol-gel method, sodium alginate polymer electrolyte using leaf extract of *Withania somnifera* through solution casting technique are briefly discussed. The basic, advanced sophisticated characterization techniques of nanoparticles such as UV-Vis, Dynamic Light Scattering (DLS) measurement, FTIR, SEM, HRTEM, XRD, XPS, DTA/TGA, and VSM analysis are discussed with a schematic diagram. In addition, the fabrication of active electrodes using synthesized NPs by doctor blade technique and working of an electrochemical workstation with instrumental setup, its characterization techniques are briefly discussed. The method of refinement techniques is explained. AC impedance spectral analysis of *Withania somnifera* leaf extract [WSLE] incorporation sodium alginate polymer electrolyte used instrumental setup also noted in this chapter. The detailed characterization results are used to understand the optical, hydrodynamic, functional group, morphological, structural, thermal, and magnetic properties of prepared materials. The studied results are more suitable for Supercapacitor and biological performance.

Chapter 4 – Electrochemical analysis of Green Synthesized Cu_2O and Ag Nanoparticles Using *Datura Metel L.*

Chapter 4 deals with synthesize Cu_2O NPs and Ag NPs using *Datura metel L*. and analyzed by its various experimental studies. The synthesized

nanoparticles are characterized by UV-Vis, FT-IR, SEM, and XRD analysis. UV-Vis results suggested the presence of hyoscyamine, and atropine in *Datura metel L*, and also, nanoparticles formation has been confirmed by the presence of absorption peak at 790 nm for Cu_2O NPs and 410 nm Ag NPs. SEM image reveals the marigold flower-like and clastic rock morphology of Cu_2O and Ag NPs. FT-IR was also used to confirm the different functional groups. The average crystallite size 19.56 nm (Cu_2O NPs) and 40 nm (Ag NPs) were obtained by XRD. The electrochemical investigation was carried out for Cu_2O and Ag NPs fabricated as electrode. The calculated maximum specific capacitance of 160 F/g at scan rate of 5 mV/s for Cu_2O NPs and 267 F/g at current density of 0.5 A/g for Ag NPs. Overall the results show that the successful preparation of Cu_2O NPs with *Datura metel L.* and tested Supercapacitor performance.

Chapter 5 – Ag Nanoparticles Synthesized by *Datura Metel L.* Leaf Extract and their Electrochemical Performance

Chapter 5 describes the various concentrations of Ag NPs synthesized using DMLE. The physical and chemical properties of synthesized Ag NPs are analyzed. The UV-Vis spectra show about the peak at 380 nm and 258 nm indicates the atropine, and hyoscyamine from DMLE along with the characterization of Ag NPs. Particle size analysis with zeta potential values 117 nm, -27.3 mV are implied that hydrodynamic size with negative surface charges. The amide functional group is the primary responsibility for the reduction of Ag NPs identified in the FTIR spectrum. SEM images reveal the spherical morphology of synthesized Ag NPs and its XRD pattern confirmed the crystalline nature. The structure factors in a single unit cell are studied as three, two, and one-dimensional profiles using the Rietveld refinement technique. (XPS) analysis display Ag 3d, Ag 3p, and Ag 3s chemical states of Ag NPs. The green synthesized Ag NPs are prepared the electrode exhibits a specific capacitance of 825 F/g at the current density of 3 mA/g in 1M KOH

electrolyte solution and cyclic stability is retained at about 88.35 % even after 5000 cycles with the current density of 5 A/g. The results showed their utilization of green synthesized Ag NPs effectively on electrochemical storage applications.

Chapter 6 - Electrochemical Investigations of *Withania Somnifera* Leaf Extract Mediated Ag/NiO Nanocomposite Symmetric, Asymmetric Supercapacitor Devices

Chapter 6 described symmetric, asymmetric Supercapacitor device performance of Ag/NiO nanocomposite synthesized via green sol-gel route using WSLE. Synthesized Ag/NiO nanocomposite characterized by several analytical techniques. UV-Vis spectra show distinct absorption peaks at 295 nm and 423 nm confirming the formation of Ag and NiO. The hydrodynamic size is 156.6 nm and zeta potential values 1.52 mV to examine the stability of the nanocomposite. The morphological studies (SEM, HRTEM) reveal the mingled hexagonal, spherical structure of Ag/NiO. The crystal structure of Ag/NiO was confirmed by XRD analysis and their structural parameters are studied through Rietveld refinement techniques. FTIR identified the alkaloids as the main compound responsible for the reduction of Ag and NiO. The presence of Ag, Ni, and O was identified in XPS analysis. The good thermal stability and superparamagnetic properties of the synthesized sample are analyzed by TGA/DTA and VSM measurement. As prepared Ag/NiO electrode exhibits specified capacitance of 872 F/g at a scan rate of 5 mV/s and exhibits good cyclic retention of about 89.28 % even after 5000 cycles. Furthermore, the admirable electrochemical properties of Ag/NiO brings the Symmetric Supercapacitor (SSC) device with an energy density of 26 Wh/kg and power density of 1580 W/kg and the asymmetrical Supercapacitor (ASC) device with an energy density of 46 Wh/kg and power density of 3501 W/kg prospects the utilization of biomass derivable electrode material for energy storage application.

Chapter 7 - Electrical and Electrochemical Characteristics of *Withania Somnifera* Leaf Extract Incorporation Sodium Alginate Polymer Film for Energy Storage Applications

Chapter 7 deals with the alternating current electrical properties and electrochemical characteristics of *Withania somnifera* leaf extract incorporated sodium alginate [WLISA] polymer film prepared under the solution casting method. Two absorption maximums (λ_{max}) were obtained for the both WLISA and *Withania somnifera leaf extract* [WSLE] samples in the UV-Vis spectrum. The XRD pattern reveals about the crystallinity along with the amorphous nature of sodium alginate that emerged due to the incorporation of WSLE. The more amount of strain that occurred in crystallization provides broaden peak in the XRD pattern. With aid of guluronic and mannuronic acids in sodium alginate making hydroxyl bond formation with leaf extract due to the cross-linking process can be analyzed from ATR-FTIR. The prepared 5 mL leaf extract WLISA film has demonstrated the highest ionic conductivity in the typical value of 2.80×10^{-6} S/Cm at room temperature. The electrochemical window of the prepared polymer film is in the range of -0.4 to 0.4 V suggesting that used as an electrolyte for Supercapacitor applications.

Chapter 8 – Summary and Conclusion

This chapter deals about the overall summary and conclusion of this thesis work. As nanoparticles, analyzed physical and chemical properties from chapters 4 to 7 are addressed. The electrochemical results of the build electrode using synthesized nanoparticles Cu_2O NPs, Ag NPs and Ag/NiO are summarised. The Ag/NiO nanocomposite mediated by the WSLE possessed calculated maximum specific capacitance compared to Ag and Cu_2O electrodes. Hence, fabricated Ag/NiO nanocomposite symmetrical, asymmetrical Supercapacitor devices are highlighted. This electrochemical performance enhanced by organic molecules present in the DMLE and WSLE is explained and the suitability of the WSLISA polymer electrolyte for

Supercapacitor devices is also discussed in the conclusion part. Moreover, synthesized nanoparticles mediated by the DMLE and WSLE as electrode material, and polymer electrolytes are useful for the fabrication of symmetric and asymmetric Supercapacitor devices and energy storage devices. The scope of the future research work continued from this thesis work is also given in this chapter.

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